NMR Measurements

for Solid Polarized Targets

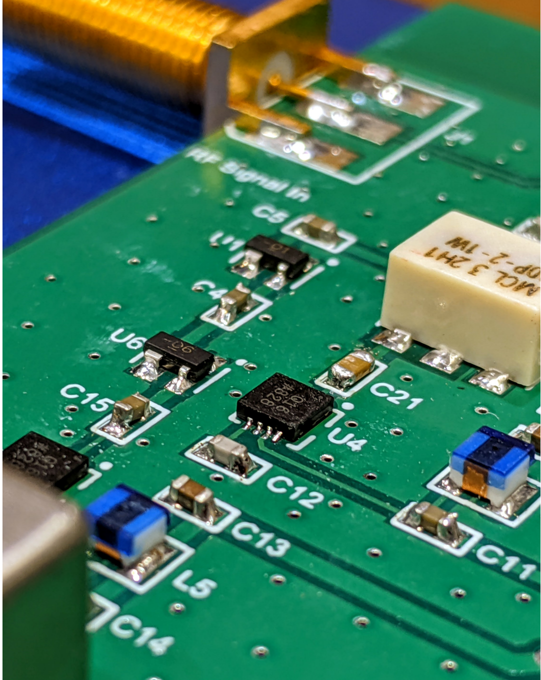
J. Maxwell

for the Jefferson Lab Target Group

Jefferson Lab

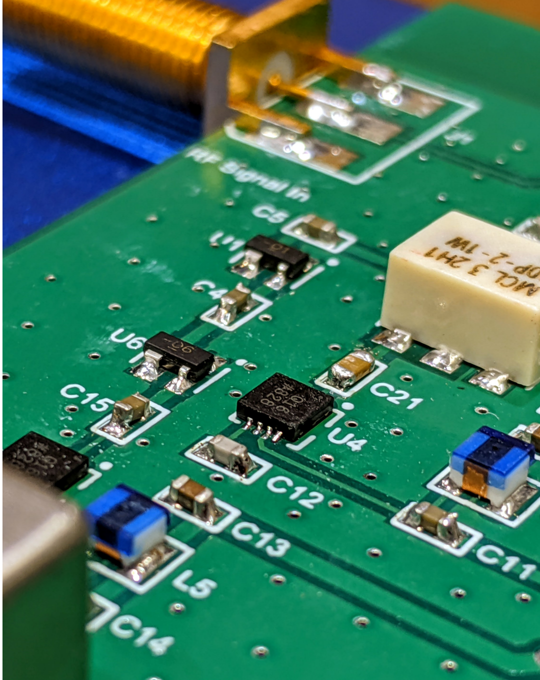
U.S. DEPARTMENT OF ENERGY

Polarized Sources, Targets and Polarimetry
Knoxville, TN
September 23rd, 2019



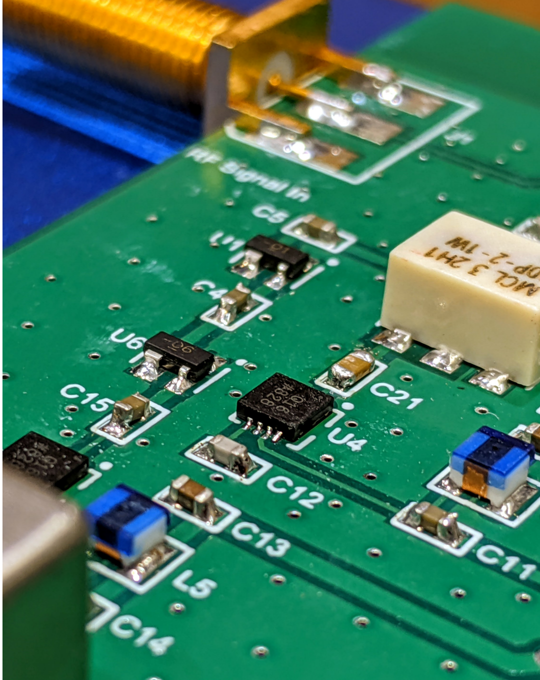
Outline

1. NMR and JLab Solid Targets
   - Introduction
   - New Challenges
2. Cold Board NMR
   - A Cautionary Tale
3. New JLab Q-Meter
   - Prototype Design
   - Initial Results



Outline

1. NMR and JLab Solid Targets
   - Introduction
   - New Challenges
2. Cold Board NMR
   - A Cautionary Tale
3. New JLab Q-Meter
   - Prototype Design
   - Initial Results



Measuring Polarization in Polarized Solids

- NMR in field B_0 at ω_0, apply RF field to material
- Coil of L_0 applies field perpendicular to B_0 to induce spin flip
- Material polarization modifies the effective inductance of a coupled coil, with filling factor η:

$$L(\omega) = L_0(1 + 4\pi\eta\chi(\omega))$$

- Polarized nuclei give the target material a complex susceptibility, a function of applied frequency (ω):

$$\chi(\omega) = \chi'(\omega) + i\chi''(\omega) \quad \text{and} \quad P = K\int_0^\infty \chi''(\omega)d\omega$$

- $\chi(\omega)$ is non-zero only close to the Larmor frequency ω_0

Measuring Polarization in Polarized Solids

- NMR in field B_0 at ω_0, apply RF field to material
- Coil of L_0 applies field perpendicular to B_0 to induce spin flip
- Material polarization modifies the effective inductance of a coupled coil, with filling factor η:

$$L(\omega) = L_0(1 + 4\pi\eta\chi(\omega))$$

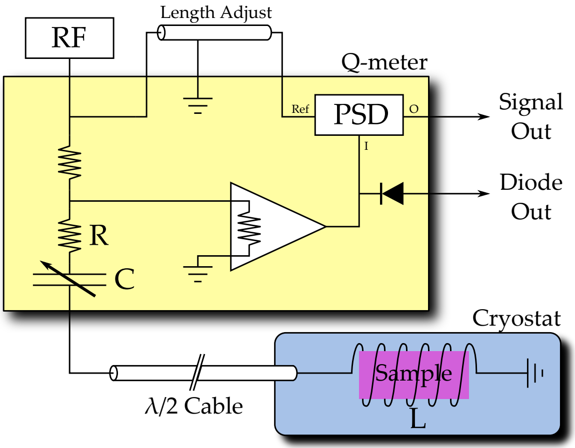
- Polarized nuclei give the target material a complex susceptibility, a function of applied frequency (ω):

$$\chi(\omega) = \chi'(\omega) + i\chi''(\omega) \quad \text{and} \quad P = K\int_0^\infty \chi''(\omega)d\omega$$

- $\chi(\omega)$ is non-zero only close to the Larmor frequency ω_0

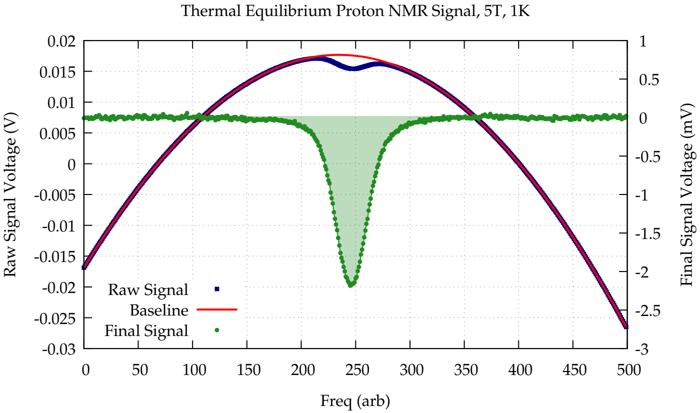
Continuous-wave NMR Electronics: Q-meter

- Choose L, C: $\omega_0 = 1/\sqrt{LC}$
- Complex impedance of circuit $\sim i\omega L_0 \Delta L(\omega)$
- Compare signal to reference with mixer for real portion, must match phase
- Away from ω_0, coil impedance has reactive components, makes Q-curve
- Sweep frequency around ω_0 to integrate in ω



Continuous-wave NMR Electronics: Q-meter

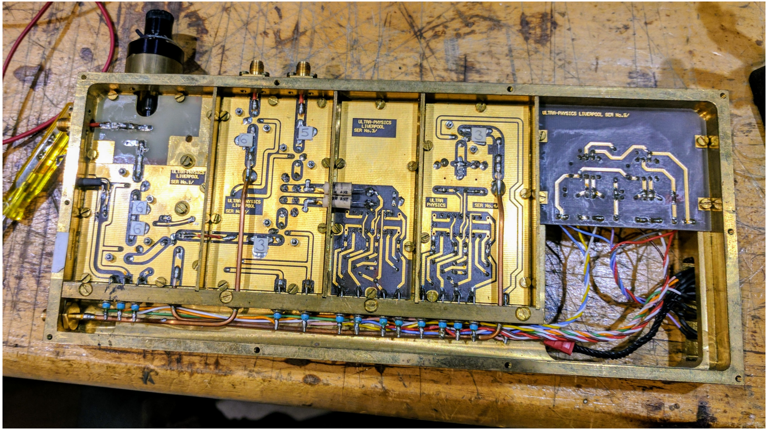
- Choose L, C: $\omega_0 = 1/\sqrt{LC}$
- Complex impedance of circuit $\sim i\omega L_0 \Delta L(\omega)$
- Compare signal to reference with mixer for real portion, must match phase
- Away from ω_0, coil impedance has reactive components, makes Q-curve
- Sweep frequency around ω_0 to integrate in ω



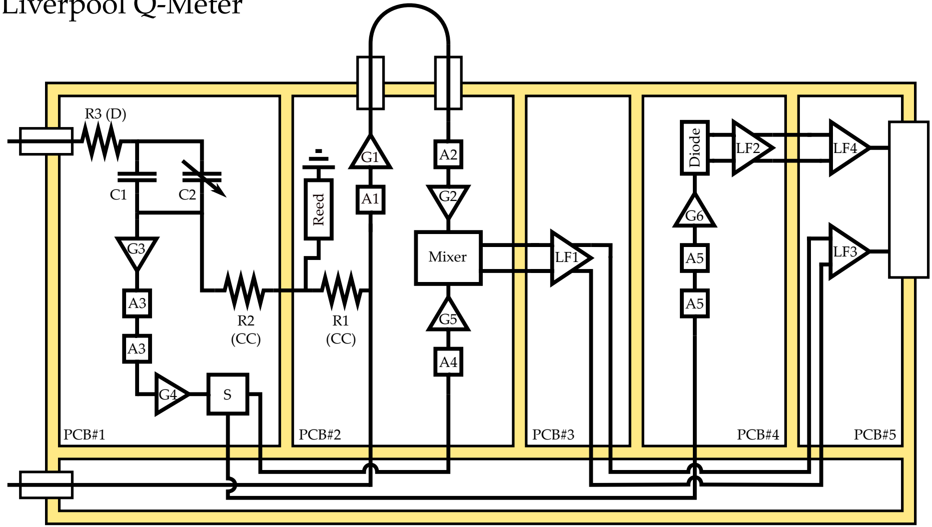
Liverpool Q-meter

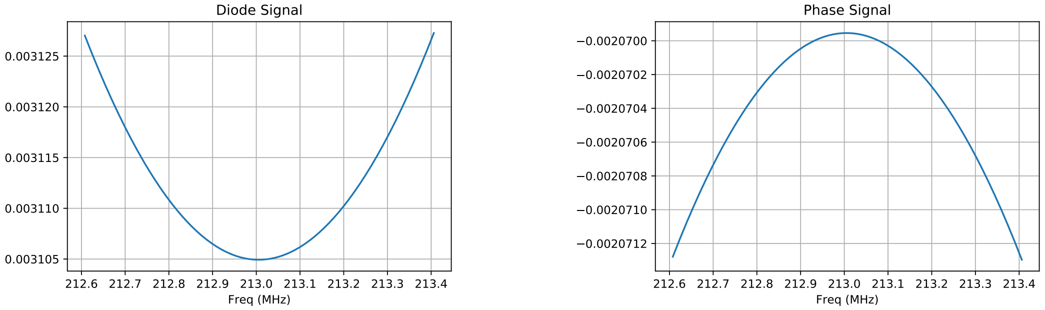
- Liverpool Q-meter used for decades.

  G.R. Court, NIM A324 (1993)
- Designed for excellent RF performance for DNP applications.
- Five main boards:
  1. Tank and splitter
  2. Mixer
  3. Phase amplification
  4. Diode amplification
  5. Final amplification



Liverpool Q-Meter



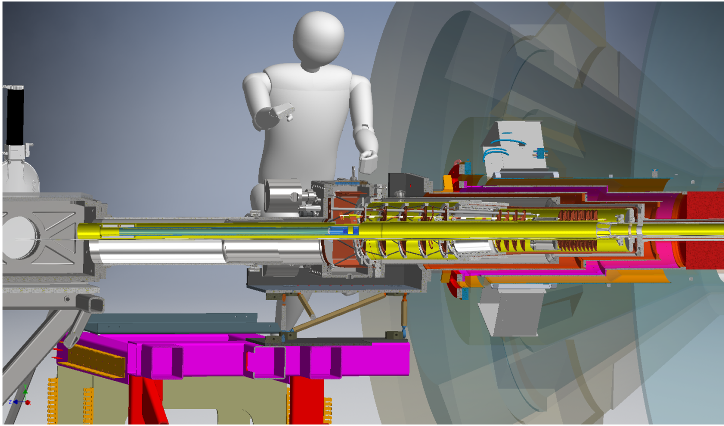


Traditional Tuning for Continuous-wave NMR

- Determine length cable $(n\lambda/2)$ to run from Q-meter to coil within cryostat
- Set C (trimmers) to center power response (diode) minimum at ω_0
- Choose phase cable to center real portion maximum at ω_0

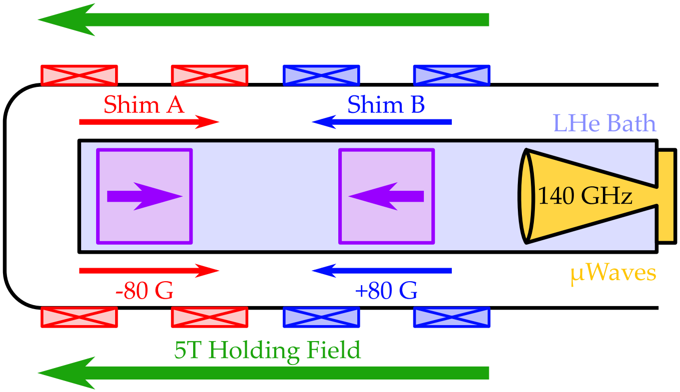
Hall B's CLAS12 Polarized Target

- External NMR coils: need high sensitivity
- Access ± polarization with $\nu_{\mu\pm} = \nu_{\text{EPR}} \pm \nu_{\text{NMR}}$ in a single holding field (COMPASS)
    - OR change local fields so that $\nu_{\mu+} = \nu_{\mu-}$
- Microwave freq static, must change shim fields, NMR tune to following change with radiation dose



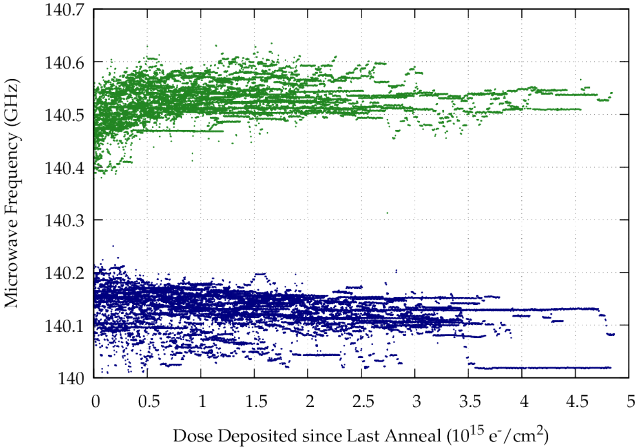
Hall B's CLAS12 Polarized Target

- External NMR coils: need high sensitivity
- Access $\pm$ polarization with $\nu_{\mu\pm} = \nu_{\text{EPR}} \pm \nu_{\text{NMR}}$ in a single holding field (COMPASS)
    - OR change local fields so that $\nu_{\mu+} = \nu_{\mu-}$
- Microwave freq static, must change shim fields, NMR tune to following change with radiation dose



Hall B's CLAS12 Polarized Target

- External NMR coils: need high sensitivity
- Access $\pm$ polarization with $\nu_{\mu\pm} = \nu_{\text{EPR}} \pm \nu_{\text{NMR}}$ in a single holding field (COMPASS)
  - OR change local fields so that $\nu_{\mu+} = \nu_{\mu-}$
- Microwave freq static, must change shim fields, NMR tune to following change with radiation dose



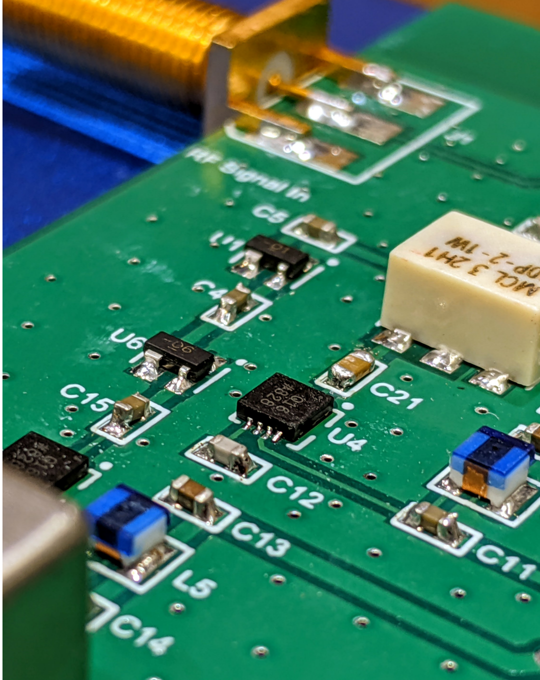
JLab NMR Wishlist

- Remote capacitor tuning
  - Accommodate 2 opposing cells, Synchronous Tuning
- Electronic phase tuning
- Cold circuit NMR
  - Noise reduction, Non-resonant cable circuit
- Address aging of Liverpool Q-meter
  - New Q-meters using off-the-shelf components

Most not revolutionary, but many previous innovations combined for the first time

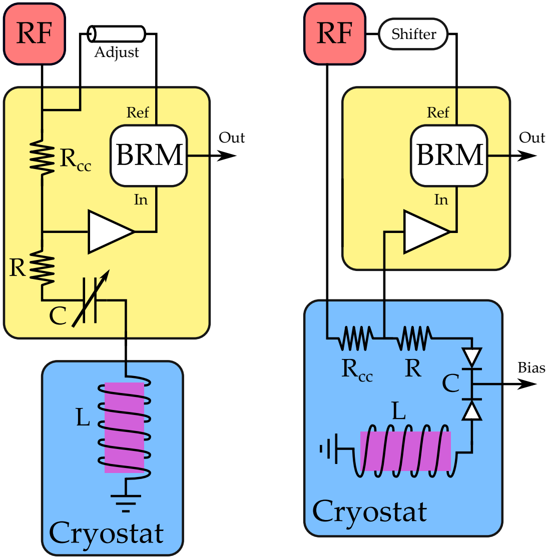
Outline

1. NMR and JLab Solid Targets
   - Introduction
   - New Challenges
2. Cold Board NMR
   - A Cautionary Tale
3. New JLab Q-Meter
   - Prototype Design
   - Initial Results



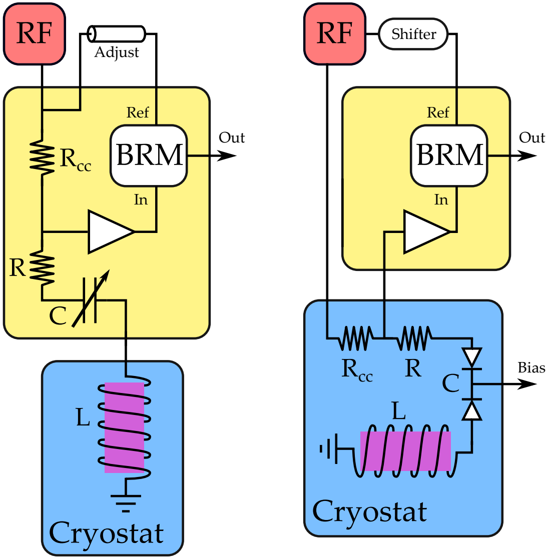
Put Tank Circuit inside Cryostat?

- Traditionally, R and C inside Q-meter, L in the cryostat
- Moving R and C into the cryostat, $\lambda/2$ no longer separates L. (Court, NIM A 2004.)
    - Q-curve shallower
    - Thermal noise reduction
    - Requires components that can handle cold, microwaves, radiation
- Varactor diodes vary C vs. voltage
- Electronic phase shifters replace phase cable
- GaAs Varactors for cryogenic applications



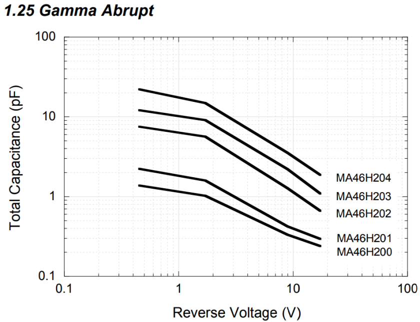
Put Tank Circuit inside Cryostat?

- Traditionally, R and C inside Q-meter, L in the cryostat
- Moving R and C into the cryostat, $\lambda/2$ no longer separates L. (Court, NIM A 2004.)
  - Q-curve shallower
  - Thermal noise reduction
  - Requires components that can handle cold, microwaves, radiation
- Varactor diodes vary C vs. voltage
- Electronic phase shifters replace phase cable
- GaAs Varactors for cryogenic applications



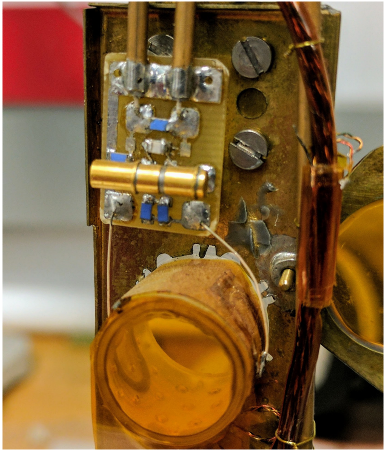
Put Tank Circuit inside Cryostat?

- Traditionally, R and C inside Q-meter, L in the cryostat
- Moving R and C into the cryostat, $\lambda/2$ no longer separates L. (Court, NIM A 2004.)
  - Q-curve shallower
  - Thermal noise reduction
  - Requires components that can handle cold, microwaves, radiation
- Varactor diodes vary C vs. voltage
- Electronic phase shifters replace phase cable
- GaAs Varactors for cryogenic applications



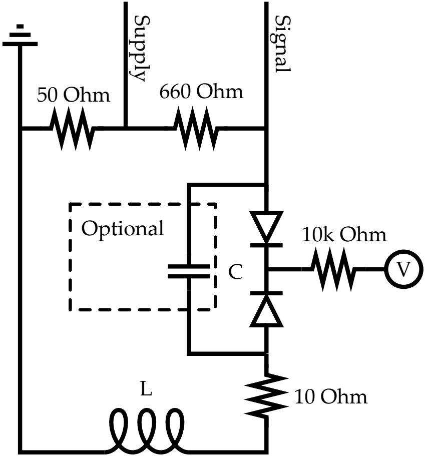
Cold NMR Boards

- Cold NMR method most recently used at JLab for EG1-DVCS deuteron
  - Trim cap and high C for 5 T D: 32.7 MHz
  - Tune performed warm, anticipating the change with temperature
- Introducing GaAs varactor diodes
  - Tested at 213 MHz to 3.2 K
  - Minimal changes seen at high B, low T



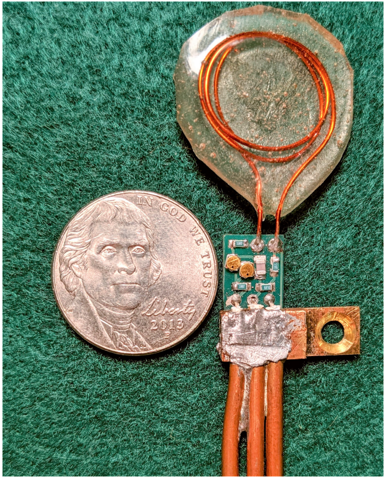
Cold NMR Boards

- Cold NMR method most recently used at JLab for EG1-DVCS deuteron
  - Trim cap and high C for 5 T D: 32.7 MHz
  - Tune performed warm, anticipating the change with temperature
- Introducing GaAs varactor diodes
  - Tested at 213 MHz to 3.2 K
  - Minimal changes seen at high B, low T



Cold NMR Boards

- Cold NMR method most recently used at JLab for EG1-DVCS deuteron
  - Trim cap and high C for 5 T D: 32.7 MHz
  - Tune performed warm, anticipating the change with temperature
- Introducing GaAs varactor diodes
  - Tested at 213 MHz to 3.2 K
  - Minimal changes seen at high B, low T

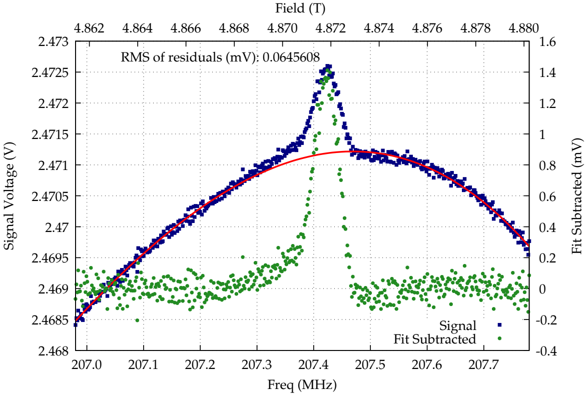


Unexpected Results

- Testing prototype Q-meters with cold tank circuit in LN bath at 5 T
- Tune Q-meter normally, see usual diode and phase curves
- Thermal Equilibrium signal appears out of Q-curve, like there is negative polarization
- Switching the phase by 180° flips, but the signal is still going the wrong way
- Observe the same behavior with Liverpool.
- Court *et al.* say if cables are matched, length doesn't matter.

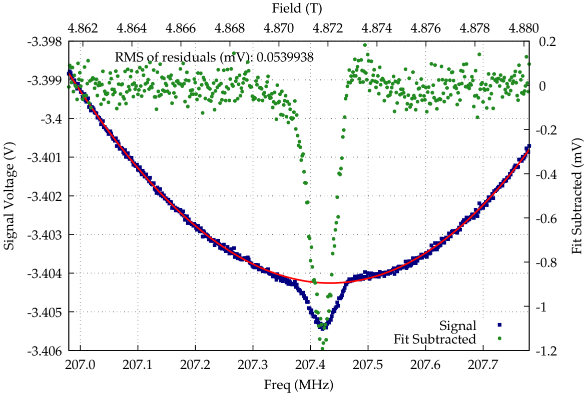
Unexpected Results

- Testing prototype Q-meters with cold tank circuit in LN bath at 5 T
- Tune Q-meter normally, see usual diode and phase curves
- Thermal Equilibrium signal appears out of Q-curve, like there is negative polarization
- Switching the phase by 180° flips, but the signal is still going the wrong way
- Observe the same behavior with Liverpool.
- Court *et al.* say if cables are matched, length doesn't matter.



Unexpected Results

- Testing prototype Q-meters with cold tank circuit in LN bath at 5 T
- Tune Q-meter normally, see usual diode and phase curves
- Thermal Equilibrium signal appears out of Q-curve, like there is negative polarization
- Switching the phase by 180° flips, but the signal is still going the wrong way
- Observe the same behavior with Liverpool.
- Court *et al.* say if cables are matched, length doesn't matter.



Unexpected Results

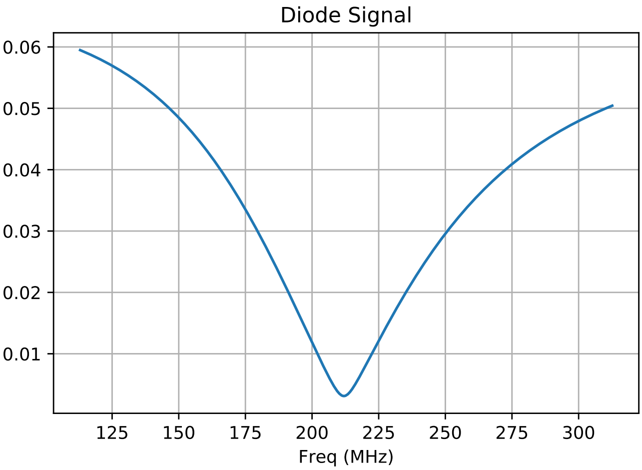
- Testing prototype Q-meters with cold tank circuit in LN bath at 5 T
- Tune Q-meter normally, see usual diode and phase curves
- Thermal Equilibrium signal appears out of Q-curve, like there is negative polarization
- Switching the phase by 180° flips, but the signal is still going the wrong way
- Observe the same behavior with Liverpool.
- Court *et al.* say if cables are matched, length doesn't matter.

5.6. Q-meter without resonant length cable

5.6.1. Principle of operation

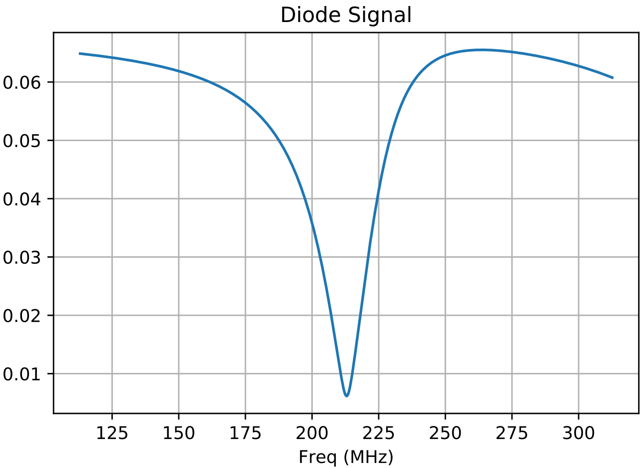
It may be concluded from the previous section that many of the operational problems associated with the standard *Q*-meter system originate with the resonant length cable. Its use can be avoided if all the passive components associated with the resonant circuit are mounted in the cold region of the target in close proximity to the coil, as shown diagrammatically in Fig. 3. Two cables are required with this arrangement, the first providing the RF feed to the coil and the second the signal feed to the voltmeter. Both are matched so their lengths are not critical. The specification of the passive components is however critical.[2] They must have good RF characteristics combined with a small temperature coefficient in the temperature range 1–6 K and low sensitivity to value change caused by microwave irradiation and beam induced radiation damage effects.

- Usually cable length matters, quite visible in tank response
- $\lambda/2$ for impedance matching between RC and L
  - 213 MHz: $\lambda/2 \sim 49$ cm
- Cold tank circuit tank has 2 long transmission cables
- Effect comes from varied lengths (J. Pierce)
- Python simulation expanded from MathCAD code by Houlden, Court.



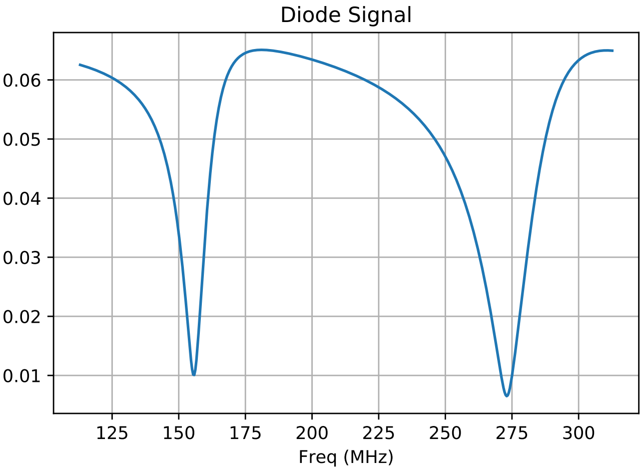
Coil cable: short

- Usually cable length matters, quite visible in tank response
- $\lambda/2$ for impedance matching between RC and L
    - 213 MHz: $\lambda/2 \sim 49$ cm
- Cold tank circuit tank has 2 long transmission cables
- Effect comes from varied lengths (J. Pierce)
- Python simulation expanded from MathCAD code by Houlden, Court.



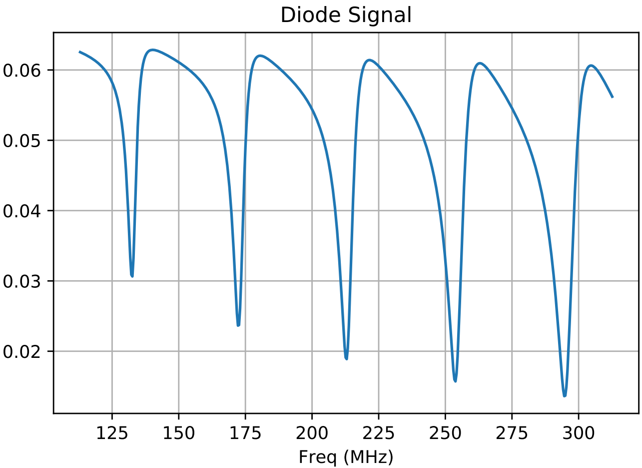
Coil cable: $\lambda/2$

- Usually cable length matters, quite visible in tank response
- $\lambda/2$ for impedance matching between RC and L
  - 213 MHz: $\lambda/2 \sim 49$ cm
- Cold tank circuit tank has 2 long transmission cables
- Effect comes from varied lengths (J. Pierce)
- Python simulation expanded from MathCAD code by Houlden, Court.



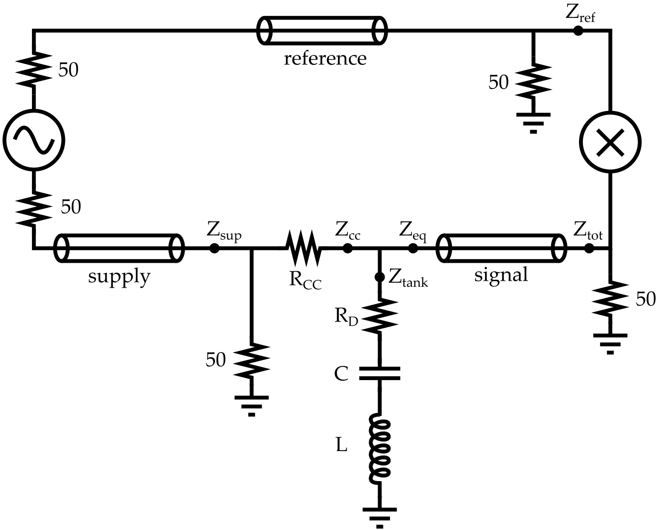
Coil cable: $1.5 \cdot \lambda/2$

- Usually cable length matters, quite visible in tank response
- $\lambda/2$ for impedance matching between RC and L
    - 213 MHz: $\lambda/2 \sim 49$ cm
- Cold tank circuit tank has 2 long transmission cables
- Effect comes from varied lengths (J. Pierce)
- Python simulation expanded from MathCAD code by Houlden, Court.

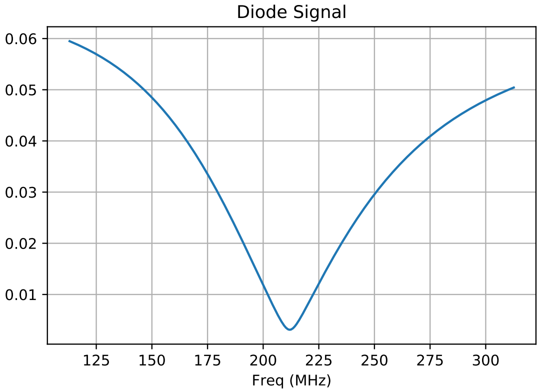


Coil cable: $5 \cdot \lambda/2$

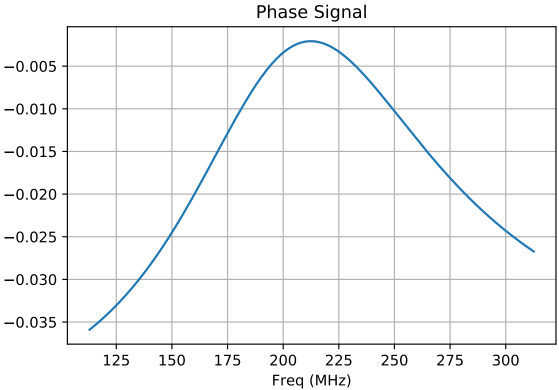
- Usually cable length matters, quite visible in tank response
- $\lambda/2$ for impedance matching between RC and L
  - 213 MHz: $\lambda/2 \sim 49$ cm
- Cold tank circuit tank has 2 long transmission cables
- Effect comes from varied lengths (J. Pierce)
- Python simulation expanded from MathCAD code by Houlden, Court.



Cold Tank Curve Response, 100 MHz Range

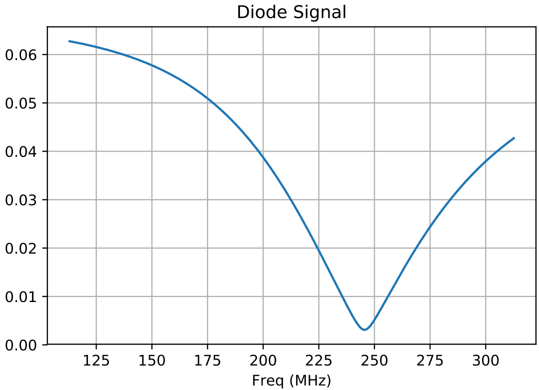


Signal cable: short

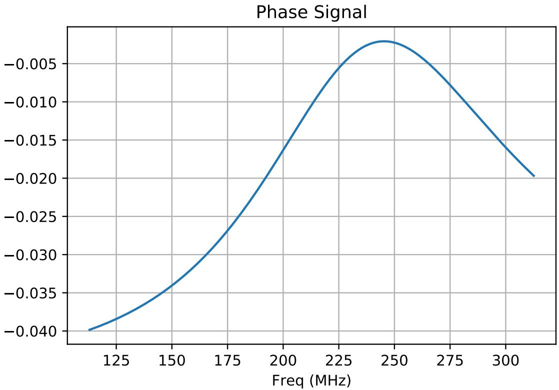


Signal cable: short

Cold Tank Curve Response, 100 MHz Range

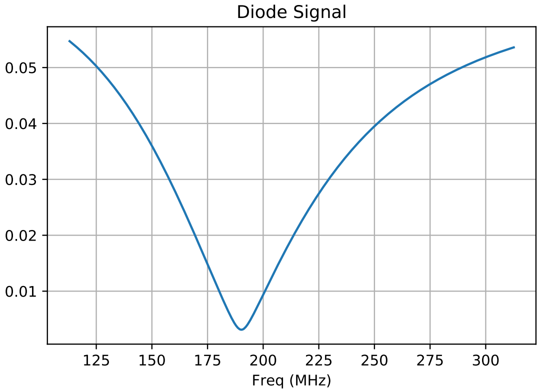


Capacitance lower

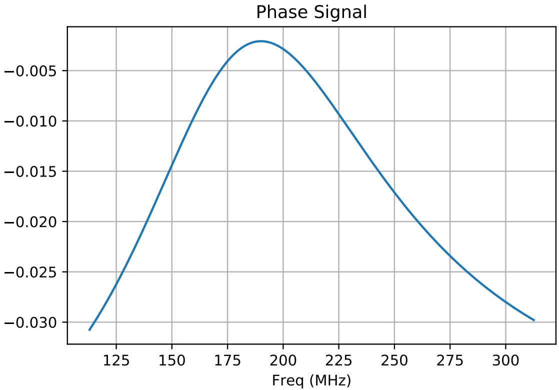


Capacitance lower

Cold Tank Curve Response, 100 MHz Range

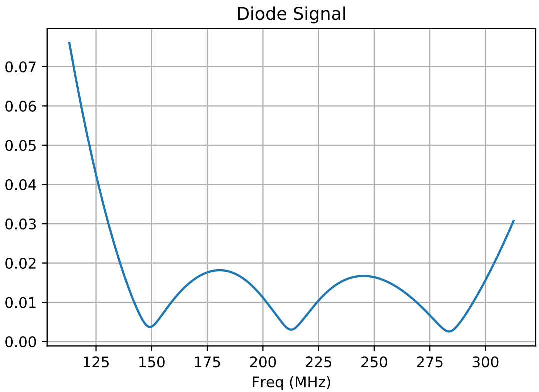


Capacitance higher

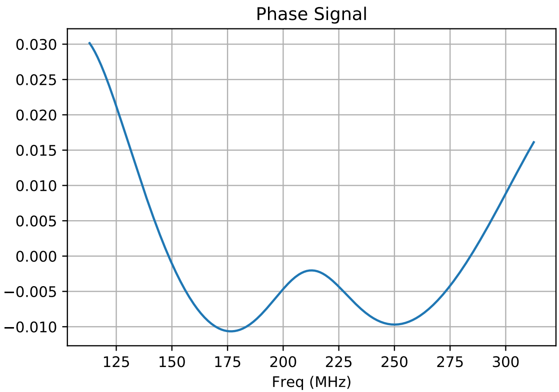


Capacitance higher

Cold Tank Curve Response, 100 MHz Range

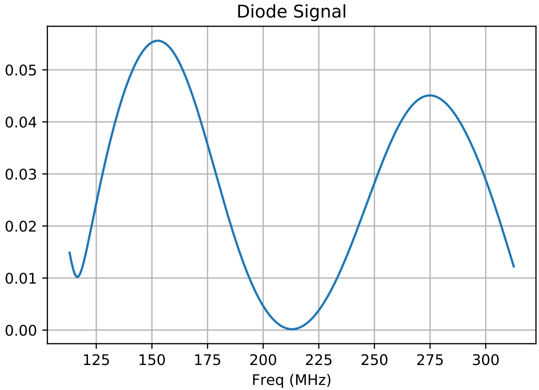


Signal cable: $\lambda/2$

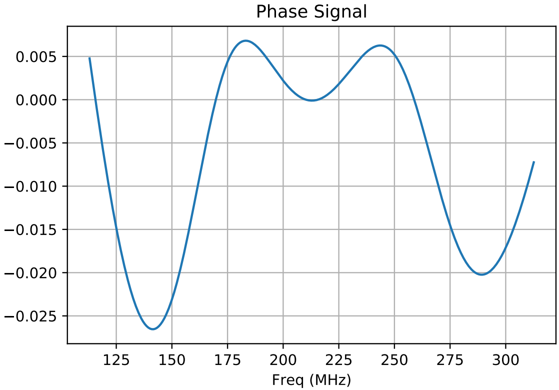


Signal cable: $\lambda/2$

Cold Tank Curve Response, 100 MHz Range

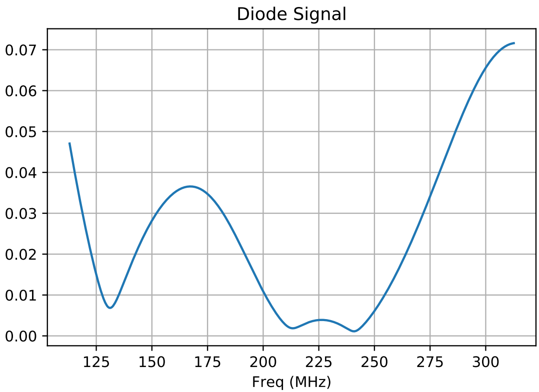


Signal cable: $1.5 \cdot \lambda/2$

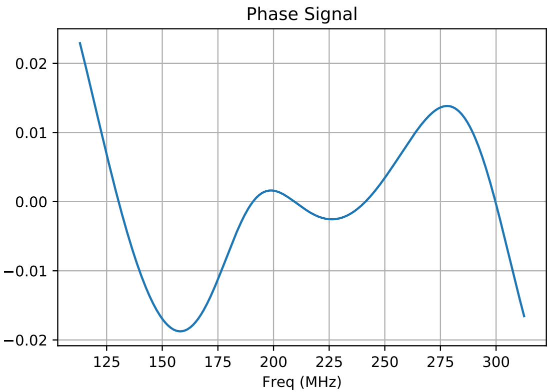


Signal cable: $1.5 \cdot \lambda/2$

Cold Tank Curve Response, 100 MHz Range

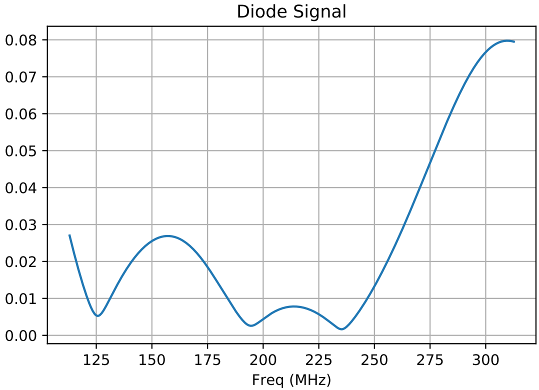


Signal cable: $1.25 \cdot \lambda/2$

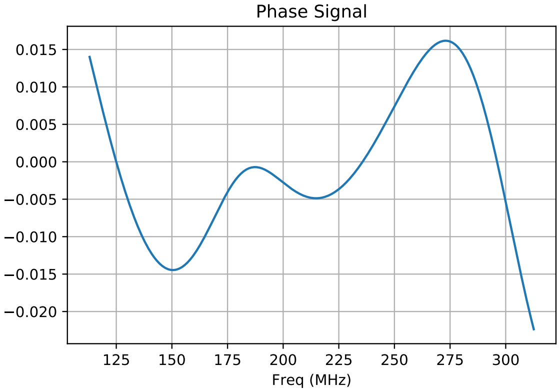


Signal cable: $1.25 \cdot \lambda/2$

Cold Tank Curve Response, 100 MHz Range

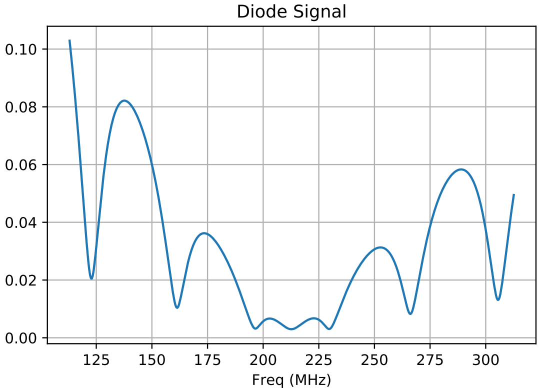


Signal cable: $1.25 \cdot \lambda/2$, retuned

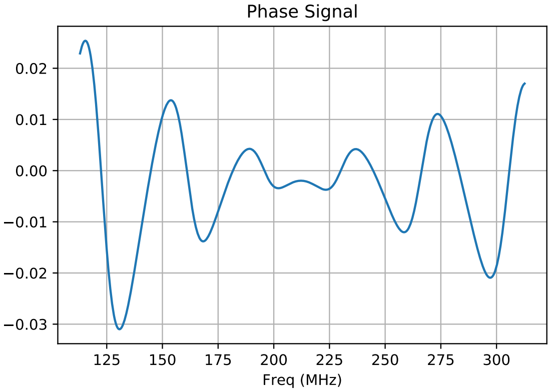


Signal cable: $1.25 \cdot \lambda/2$, retuned

Cold Tank Curve Response, 100 MHz Range

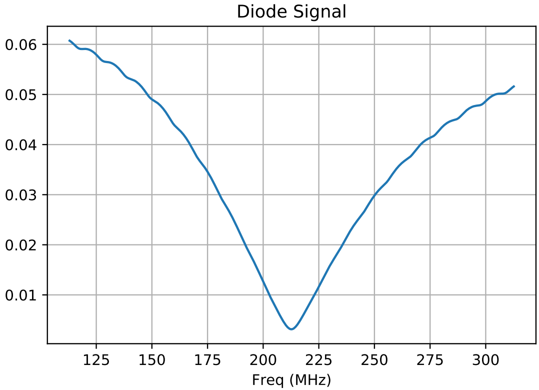


Signal cable: $5 \cdot \lambda/2$

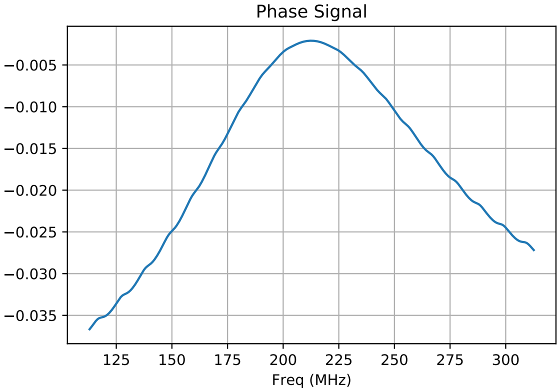


Signal cable: $5 \cdot \lambda/2$

Cold Tank Curve Response, 100 MHz Range

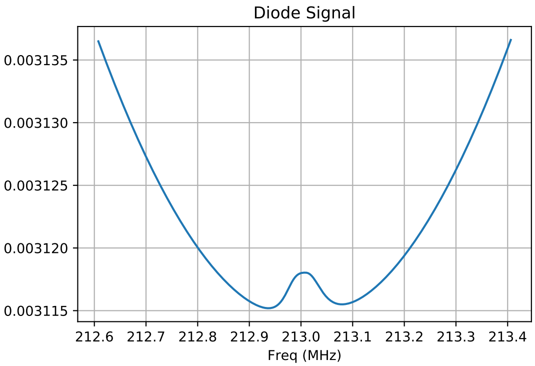


Supply cable: $20 \cdot \lambda/2$

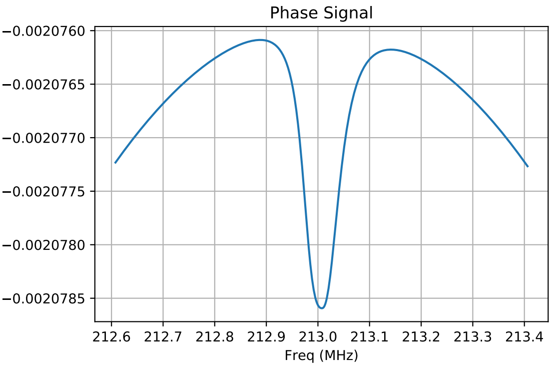


Supply cable: $20 \cdot \lambda/2$

Q-curve and Signal Response, 400 kHz Range

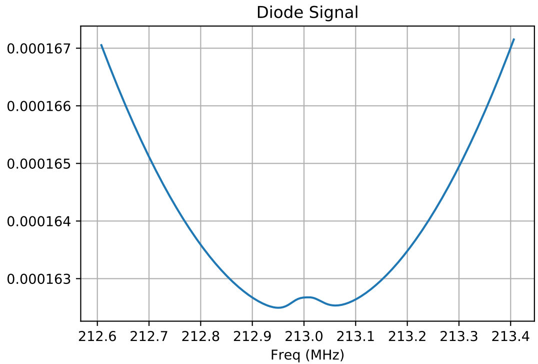


Signal cable: short

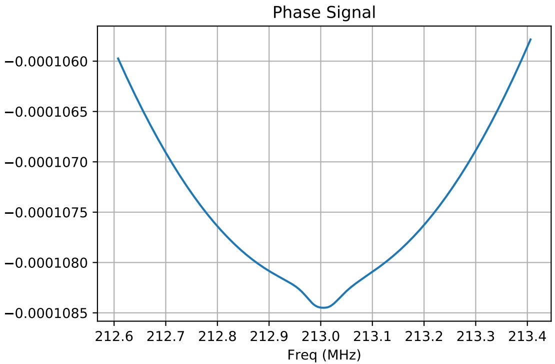


Signal cable: short

Q-curve and Signal Response, 400 kHz Range

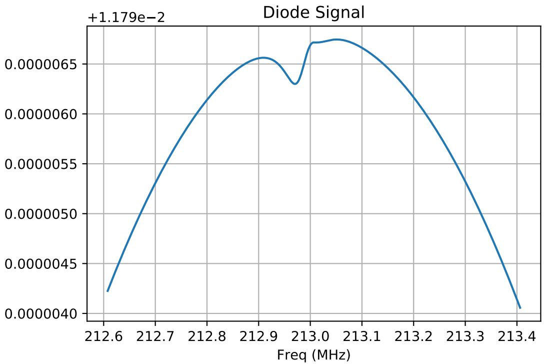


Signal cable: $1.5 \cdot \lambda/2$

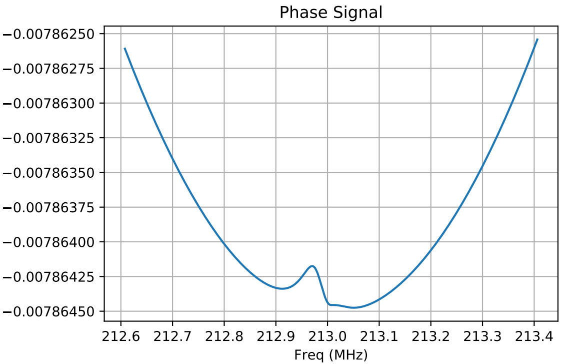


Signal cable: $1.5 \cdot \lambda/2$

Q-curve and Signal Response, 400 kHz Range



Signal cable: $1.2 \cdot \lambda/2$, retuned



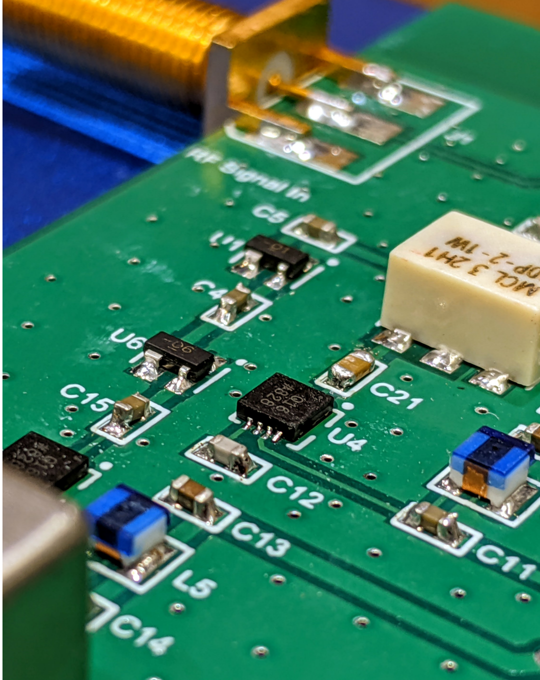
Signal cable: $1.2 \cdot \lambda/2$, retuned

Lessons

- Cable length does matter, particularly at high frequency.
  - For 32,7 MHz deuterons and 3.2 m $\lambda/2$, this effect was negligible for practical cable lengths.
  - With 213 MHz protons, 49 cm is short enough to see large effects with small length changes.
  - We need around $10\lambda/2$ just to get into the CLAS12 polarized target!
- The ease that comes with turning a dial to change the capacitance and phase has dangers.
  - Pay attention to the depth of the Q-curve and signal.

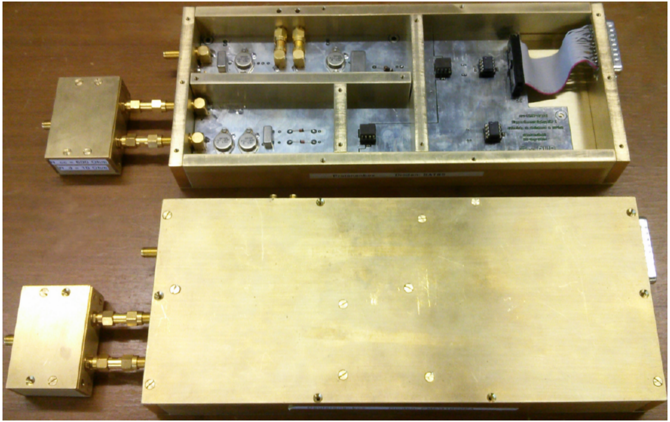
Outline

1. NMR and JLab Solid Targets
   - Introduction
   - New Challenges
2. Cold Board NMR
   - A Cautionary Tale
3. New JLab Q-Meter
   - Prototype Design
   - Initial Results



A New Liverpool Q-meter

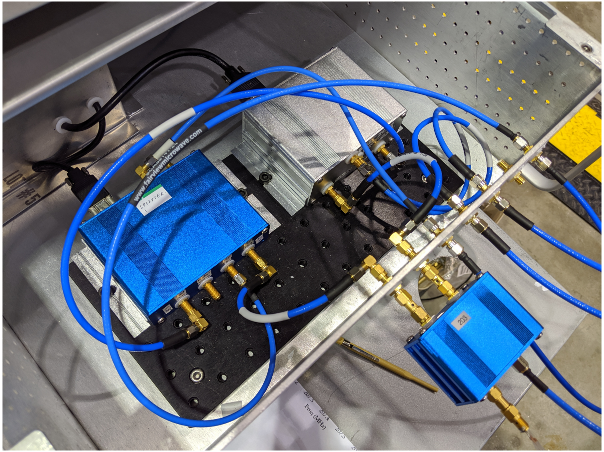
- Q-meters now limited commodity
- Bochum group already building new Q-meters in the Liverpool style with new components
- We followed their lead, adding improvements
  - Off-the-shelf components
  - Modular as possible
  - Modern amplifiers
  - Require only ± 5 V supply
- Prototype Complete



J. Herick Thesis, Bochum, 2016.

A New Liverpool Q-meter

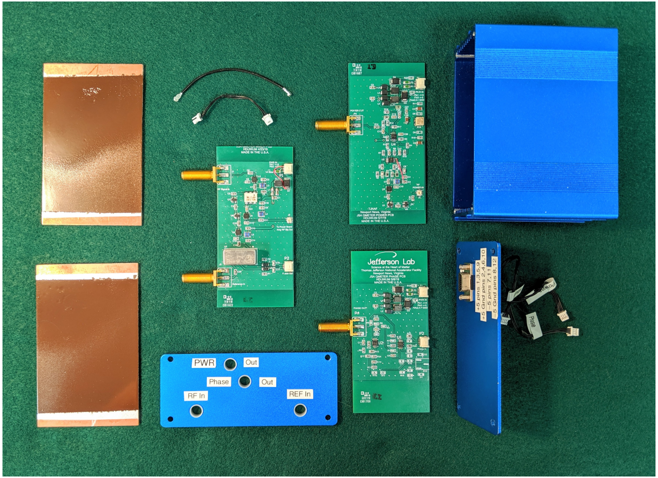
- Q-meters now limited commodity
- Bochum group already building new Q-meters in the Liverpool style with new components
- We followed their lead, adding improvements
    - Off-the-shelf components
    - Modular as possible
    - Modern amplifiers
    - Require only ± 5 V supply
- Prototype Complete



Q-meter Prototype Design

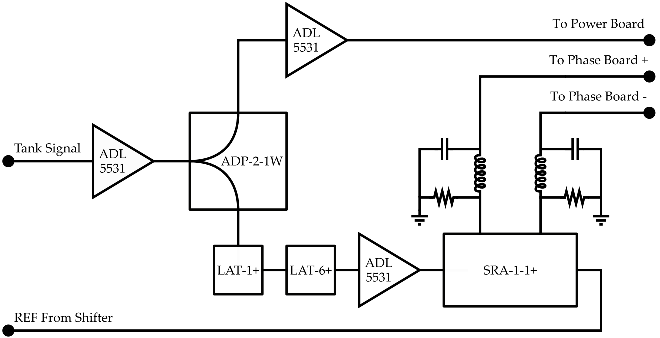
- JLab Q-meter consists of 3 boards in Al enclosure
- Boards stacked vertically with shielding between layers
- “Stitching” for ground planes
- Mixer, diodes: same as Bochum
- Biggest change: Analog Devices RF and Diff amplifiers

1. Mixer Board
2. Phase Amp
3. Diode Amp Board



Q-meter Prototype Design

- JLab Q-meter consists of 3 boards in Al enclosure
- Boards stacked vertically with shielding between layers
- “Stitching” for ground planes
- Mixer, diodes: same as Bochum
- Biggest change: Analog Devices RF and Diff amplifiers

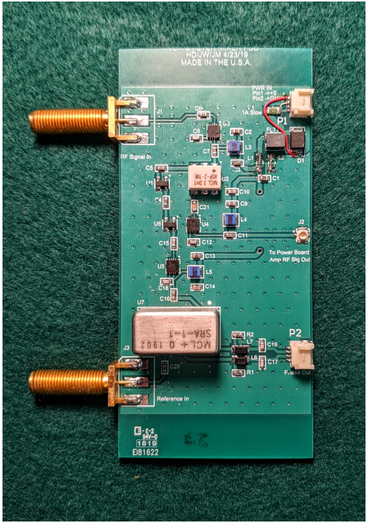


1. Mixer Board
2. Phase Amp
3. Diode Amp Board

Q-meter Prototype Design

- JLab Q-meter consists of 3 boards in Al enclosure
- Boards stacked vertically with shielding between layers
- “Stitching” for ground planes
- Mixer, diodes: same as Bochum
- Biggest change: Analog Devices RF and Diff amplifiers

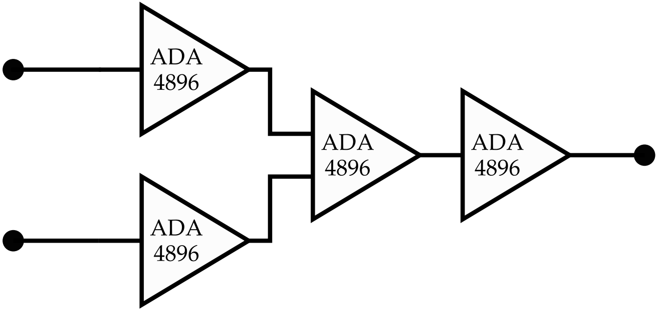
1. Mixer Board
2. Phase Amp
3. Diode Amp Board



Q-meter Prototype Design

- JLab Q-meter consists of 3 boards in Al enclosure
- Boards stacked vertically with shielding between layers
- “Stitching” for ground planes
- Mixer, diodes: same as Bochum
- Biggest change: Analog Devices RF and Diff amplifiers

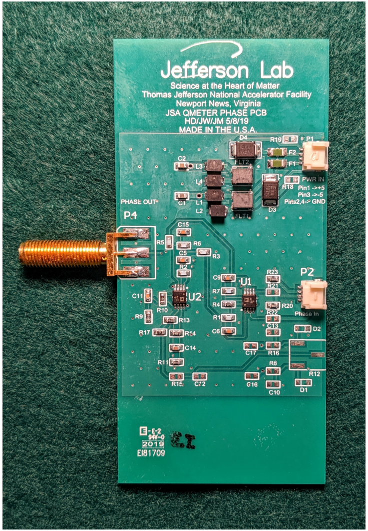
1. Mixer Board
2. Phase Amp
3. Diode Amp Board



Q-meter Prototype Design

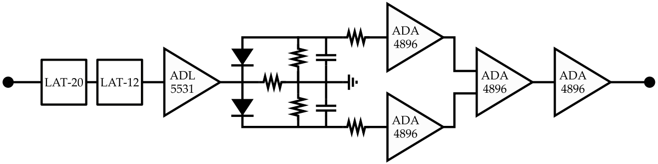
- JLab Q-meter consists of 3 boards in Al enclosure
- Boards stacked vertically with shielding between layers
- “Stitching” for ground planes
- Mixer, diodes: same as Bochum
- Biggest change: Analog Devices RF and Diff amplifiers

1. Mixer Board
2. Phase Amp
3. Diode Amp Board



Q-meter Prototype Design

- JLab Q-meter consists of 3 boards in Al enclosure
- Boards stacked vertically with shielding between layers
- “Stitching” for ground planes
- Mixer, diodes: same as Bochum
- Biggest change: Analog Devices RF and Diff amplifiers

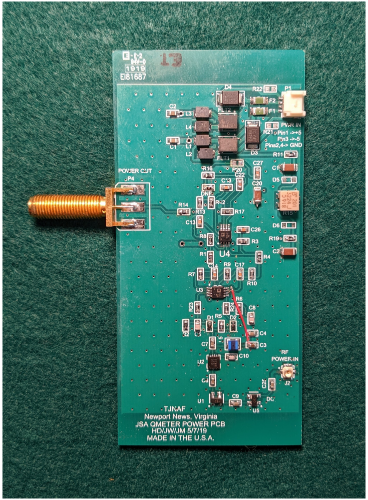


1. Mixer Board
2. Phase Amp
3. Diode Amp Board

Q-meter Prototype Design

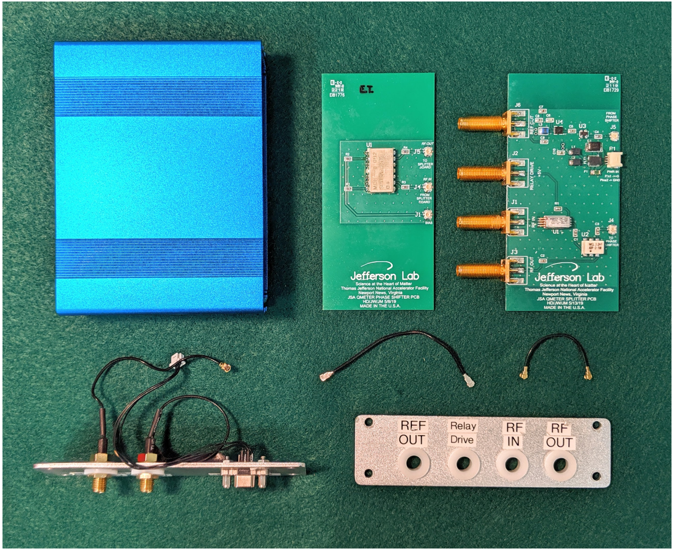
- JLab Q-meter consists of 3 boards in Al enclosure
- Boards stacked vertically with shielding between layers
- “Stitching” for ground planes
- Mixer, diodes: same as Bochum
- Biggest change: Analog Devices RF and Diff amplifiers

1. Mixer Board
2. Phase Amp
3. Diode Amp Board



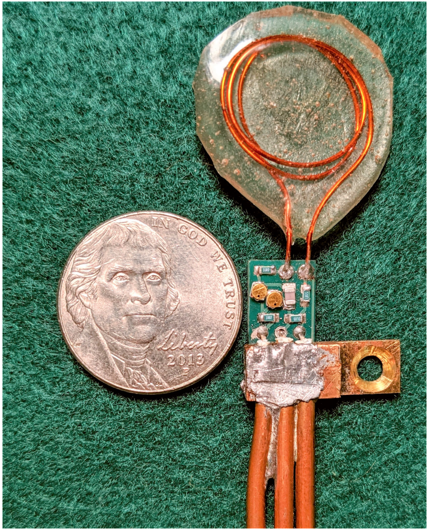
Auxiliary Box Design

- Split RF-in to send to tank board and phase shifter
- Amplify RF to send as REF to Q-meter
- Accommodate either electronic phase shifter or phase cable
- Turn off RF power to Q-meter through relay when not in use



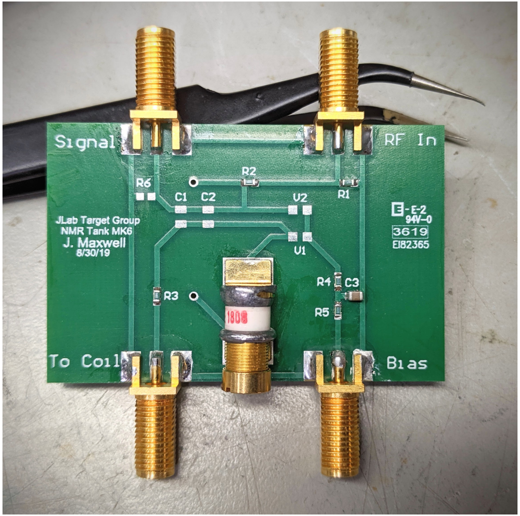
Tank Board Options

- Cold tank boards
  - Macom MA46H20x Varactors
  - Slot for 1 static cap
- External tank boards
  - Use varactors, trimmer, and/or static caps
  - Extruded Al enclosure can mount directly to chassis



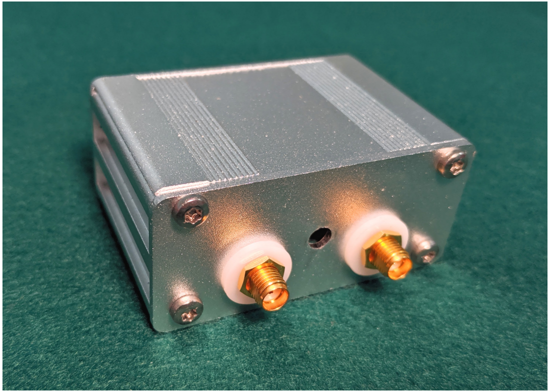
Tank Board Options

- Cold tank boards
  - Macom MA46H20x Varactors
  - Slot for 1 static cap
- External tank boards
  - Use varactors, trimmer, and/or static caps
  - Extruded Al enclosure can mount directly to chassis



Tank Board Options

- Cold tank boards
  - Macom MA46H20x Varactors
  - Slot for 1 static cap
- External tank boards
  - Use varactors, trimmer, and/or static caps
  - Extruded Al enclosure can mount directly to chassis



Cool-down Test

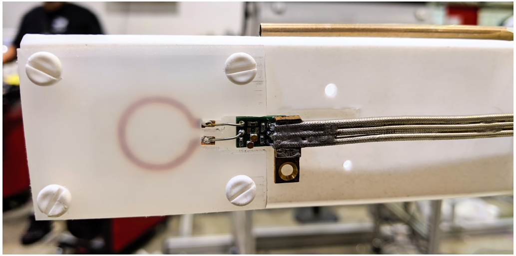
- September 16-18
- Cold tank and traditional coil installed
- Coils outside insert, away from sample
- Encountered temperature dependent structure in frequency spectrum
- Encountered intermittent background noise (ambient?)

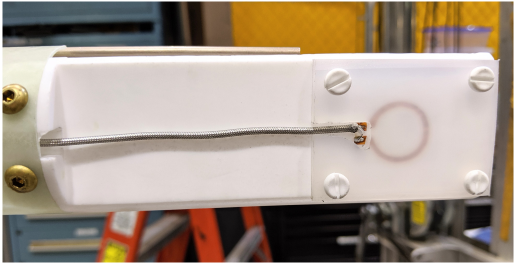
Cool-down Test

- September 16-18
- Cold tank and traditional coil installed
- Coils outside insert, away from sample
- Encountered temperature dependent structure in frequency spectrum
- Encountered intermittent background noise (ambient?)

Cool-down Test

- September 16-18
- Cold tank and traditional coil installed
- Coils outside insert, away from sample
- Encountered temperature dependent structure in frequency spectrum
- Encountered intermittent background noise (ambient?)



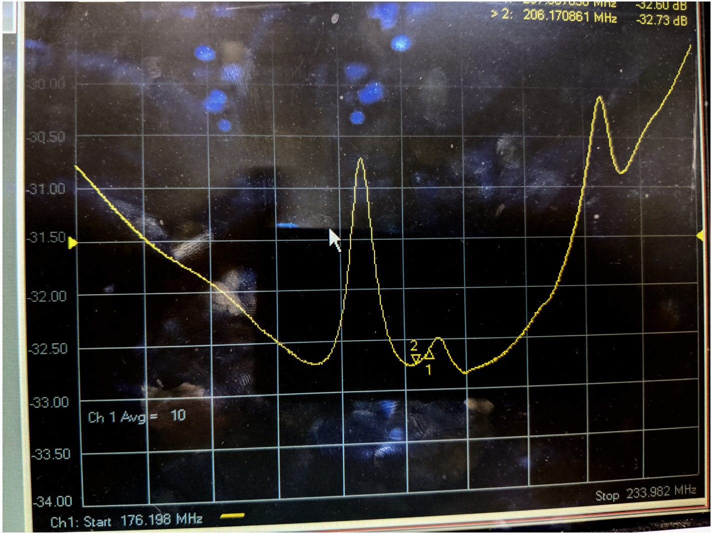


Cool-down Test

- September 16-18
- Cold tank and traditional coil installed
- Coils outside insert, away from sample
- Encountered temperature dependent structure in frequency spectrum
- Encountered intermittent background noise (ambient?)

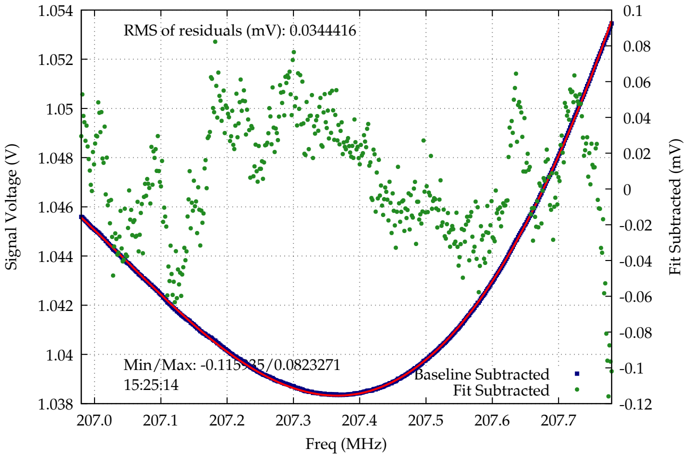
Cool-down Test

- September 16-18
- Cold tank and traditional coil installed
- Coils outside insert, away from sample
- Encountered temperature dependent structure in frequency spectrum
- Encountered intermittent background noise (ambient?)



Cool-down Test

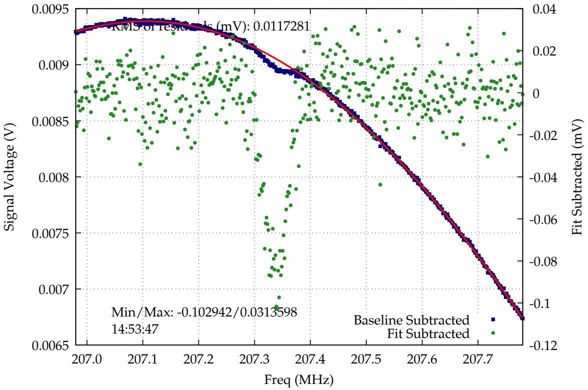
- September 16-18
- Cold tank and traditional coil installed
- Coils outside insert, away from sample
- Encountered temperature dependent structure in frequency spectrum
- Encountered intermittent background noise (ambient?)



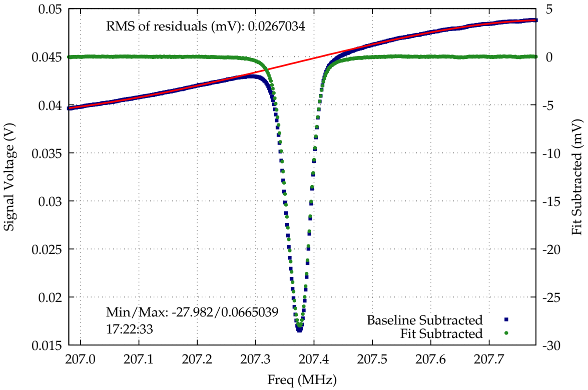
Baseline - baseline: 10,000 sweeps averaged

JLab NMR Prototype Works!

Roughly 60% polarization measured in TEMPO doped epoxy.



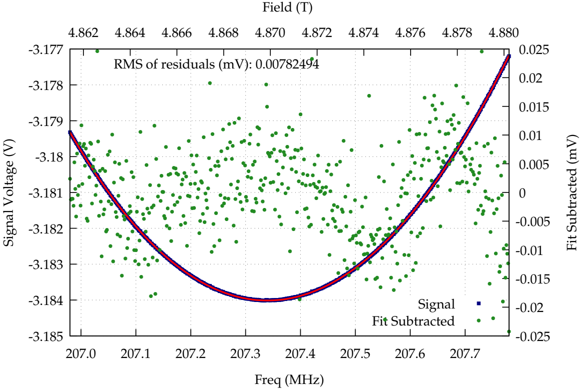
TE, 5000 sweeps



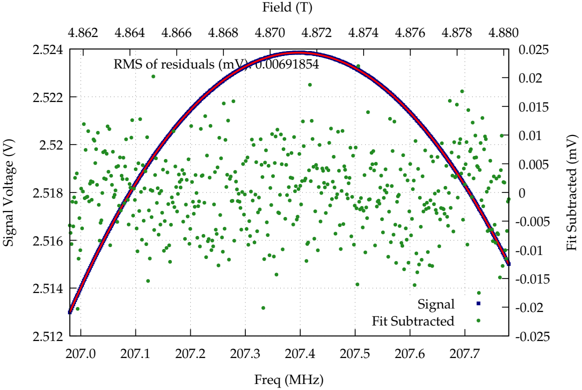
Enhanced, 2000 sweeps

Comparison to Liverpool: Noise

Noise level is comparable in same amount measuring time.



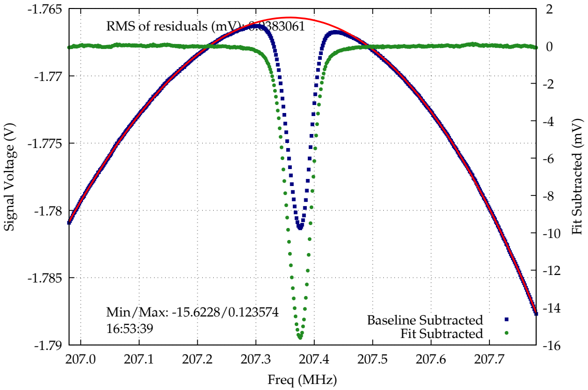
Liverpool, 10,000 sweeps averaged



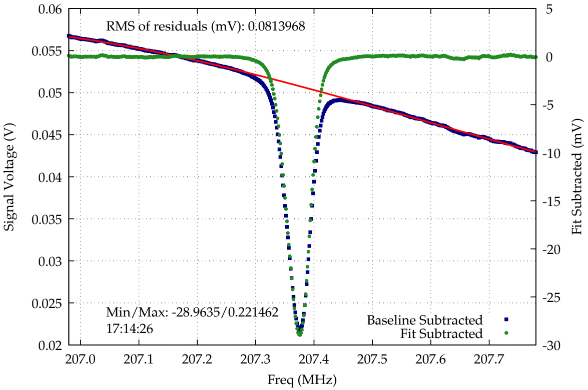
JLab, 10,000 sweeps averaged

Comparison to Liverpool: Signal Size

Max polarization, same external tank box, JLab signal nearly twice as large.



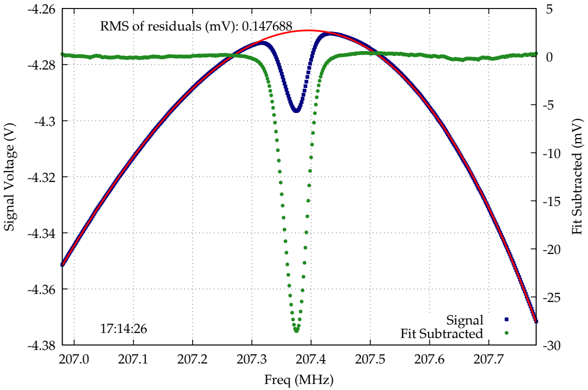
Liverpool, 1000 sweeps averaged



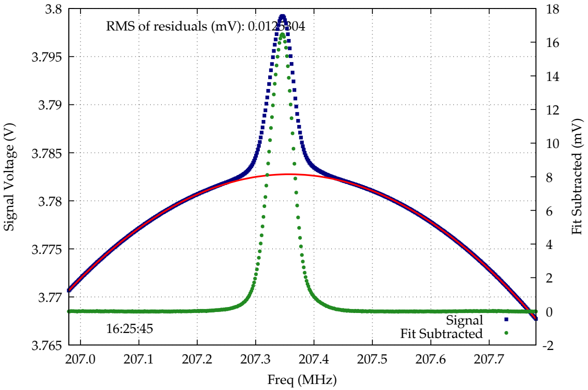
JLab, 5000 sweeps averaged

Cold Tank Circuit vs. External Tank Circuit

Cold tank signal peak wrong way. Much less susceptible to intermittent noise.



External tank circuit, 2000 sweeps averaged



Cold tank circuit, 1000 sweeps averaged

Next Steps

- Fixes and Checks
  - Noise, intermittent ambient and refrigerator-based
  - Temperature stabilization
  - Low temperature dependence and long term stability of varactors
  - Linearity checks, operating levels
  - Yale amplification card comparisons
- Improvements
  - Interface: New ADC and DAC
  - Software Improvements
  - All Digital Q-meter?

Jefferson Lab Polarized Target Group:

- C. Keith, J. Maxwell, D. Meekins
- J. Brock, C. Carlin, D. Griffith, M. Hoegerl, P. Hood

Special thanks:

- H. Dong, J. Wilson
  (JLab Electronics Group)
- J. Pierce (ORNL)
- V. Lagerquist (ODU)

Thank you for your attention!